

THE INSTITUTE SPOKESMAN



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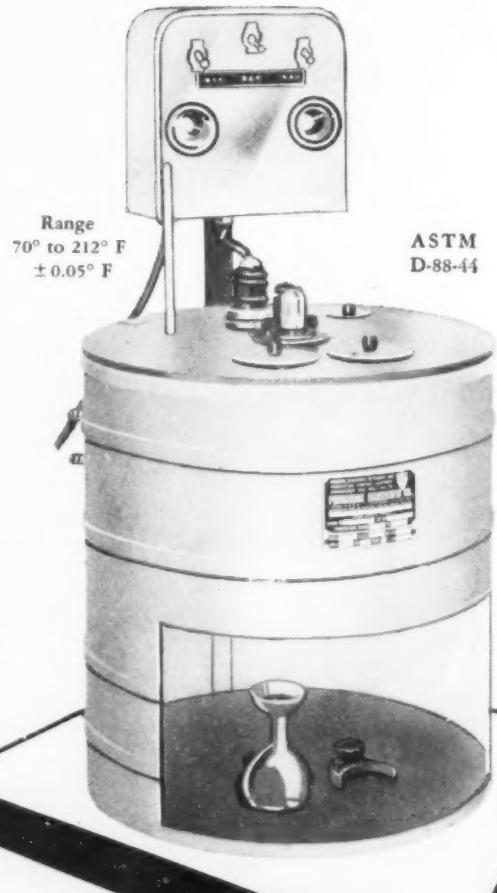
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ABOUT THE COVER

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Essentially, a wear test involves measuring the diameter of scars worn into the surface of three stationary balls by a fourth rotating ball held above and pressed against them by means of an adjustable force (load).

The points of contact of the balls are lubricated by the oil or grease under test in a cup surrounding the Four-Ball assembly. The diameter of the scars, which is a measure of the wear, is determined by means of a low power microscope.

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Technical Committee Column

This is the first issue of the Technical Committee Column. We hope that this special column will serve to keep members better informed of Committee activities by reporting them here soon after they occur rather than accumulating them for the annual meetings. In addition, it is planned to utilize this space for carrying along technical notes pertaining to grease problems. The membership of the Technical Committee includes representatives of raw material suppliers, grease manufacturers, grease dispensing equipment manufacturers and grease consumers. It is intended to encourage a flow of items from all four of those interests. The items should, of course, be brief and should not be replacements for papers. This column could develop into a convenient means for carrying on an open forum

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THE NATIONAL LUBRICATING
GREASE INSTITUTE

CARL E. BOLTE *Editor*
4638 Millcreek Parkway
Kansas City 2, Mo.

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larly grease guns, in terms of physical characteristics of the greases they will handle. Under the Chairmanship of Mr. Carl Georgi, the Committee has done a good job in exploring the many factors involved and in directing attention of all parties concerned to its importance. Therefore, the next step is now under way, namely, the organization of a Panel on which both grease and dispensing equipment manufacturers will have representation. The Panel's first job will be to formulate a definite and detailed program which will cover the selection of test conditions and reference oils and greases. Full advantage will be taken of the information given in the Committee's report, entitled "The Pumpability and Delivery Characteristics of Grease Dispensing Equipment," and all data disclosed during discussions of this subject at previous meetings of the Technical Committee. Information regarding the activities of this Panel will be reported in this column at regular intervals.

The ABEC-NLGI Cooperative Committee on Grease Test Methods, as indicated by its title, undertakes projects involving grease test methods which are of mutual interest to both the anti-friction bearing and grease industries. ABEC is shorthand for Annular Bearing Engineers' Committee of the Anti-Friction Bearing Manufacturer's Association, Inc. The present membership of the Cooperative Committee is the following:

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Mr. C. R. Gillette, General Motors Corp., New Departure Division.

Mr. C. T. Hewitt, Fafnir Bearings Corporation.
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Mr. E. E. Wagner, Hoover Ball & Bearing Company.
Mr. F. L. Wright, Norma-Hoffmann Bearings Corporation.

Representatives of the Technical Committee of NLGI

Dr. E. W. Adams, Standard Oil Company (Indiana).
Mr. H. A. Ambrose, Gulf Research & Development Company (Mr. P. R. McCarthy, alternate).

Dr. C. L. Fleming, Jr., Standard Oil Development Company.

(Continued on page 11)

HARDESTY

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GREASE MANUFACTURE . . .

Mr. C. L. Johnson

Mr. C. L. Johnson has had considerable experience in the manufacture of various greases and in directing a well-known grease manufacturing company which has shown a very progressive spirit in the grease industry.

He was graduated from Oklahoma University in 1917.

He started his business career with the Maryland Refining Company in Ponca City, Oklahoma.

In 1929 he became associated with the Jesco Lubricant Company of Kansas City, with which Company he is still connected.

"Mr. Manager, may we visit your grease plant?" "Certainly not," he replies indignantly. "Greasemaking is a confidential art—its intricacies are known only by our patriarchal employee, Mr. Smith, who has been making our greases for 30 years. He and he alone knows what ingredients are necessary to make a product of given quality—he and he alone is capable of judging the quality of the raw materials, and he and he alone is capable of judging the quality of finished products. No, we the management do not have a record of the formulae. Those are in Mr. Smith's head. No, we cannot guarantee that we will ever ship two batches alike." "What would you do if Mr. Smith were to leave you?" "That's very simple, his son is his apprentice and is learning the secrets from him."

No, the grease manufacturing industry does not follow such procedures anymore—not quite that antiquated—but many of the bad habits acquired over many years of such operations are still carried into present manufacturing technique.

For example, batches are judged visually by the greasemaker for water content and alkalinity, alkalis are added in indiscriminate solution, constituents are added by volume instead of by weight, fatty acids, fats and alkali are inadequately checked in advance for quality, etc. In our opinion, compounding a batch of grease should be a science of precision, and we enumerate below some of the major considerations involved.

Manufacturing Control

Our conception of grease manufacturing is one of chemical process where various components are combined in fixed predetermined amounts by a fixed predetermined technique for the manufacture of a fixed predetermined prod-

An Art or Science?

• CLAUDE L. JOHNSON, Pres., Jesco Lubricants Co., North Kansas City, Mo., & J. ALTSCHULER, Vice-Pres., Stratford Eng. Corp., Kansas City, Mo.

Delivered before the 15th Annual N.L.G.I. Convention, Edgewater Beach Hotel, Chicago, Illinois, Oct. 16-18, 1947.



Claude L. Johnson

uct. In order to minimize non-uniformity of product and difficulties in the manufacturing process which after laboratory check must be corrected, involving loss of manhours and equipment time, it is our belief that all ingredients which enter a batch of grease should be positively and accurately controlled, both as to quantity and quality. In most conventional grease plant operations we find that this procedure is not followed due, we believe, to several causes. Some of these causes are:

- A. Inadequacy of mixing in soap kettles.
- B. Inadequacy of mixing in dehydration, blending and finishing kettles.
- C. Variation in quality of raw materials.
- D. 168-hour week operation, which makes adequate chemical supervision of greasemaking difficult, if not impossible.
- E. The fact that present installations and their operation were developed under the old belief that grease-

making is not a science, but an art known only by the artisan greasemaker, with the quality of product and steps in manufacture subject to his visual judgment.

F. Shift operation with frequent changing personnel.

(1) Pres., Jesco Lubricants Co., North Kansas City, Mo.

(2) Vice-Pres., Stratford Eng. Corp., Kansas City, Mo.

It is our recommendation that every possible effort be made to place the making of grease on a strictly chemical formula precision basis. The present method results in reduced capacity equipment and the necessity of reworking, or even loss of, many batches of grease, both of which we believe can be very largely avoided.

Supervision

The quality of any operation is only as good as its supervision. Most grease plants are operated 24 hours per day, six or seven days a week, requiring a large staff of supervisors, who, for good results, should be well versed in greasemaking. It is always a problem to have thoroughly trained and experienced personnel available around the clock. Lack of such personnel usually results in batches which must be worked over, resulting in lost time for both equipment and personnel with resultant loss of production. A logical answer to this problem



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to use only 8 hours per day, 5 days per week for manufacturing, thereby requiring only a minimum of supervisory personnel, and at all times having operations under most able control available.

Raw Materials

We all know too well the variations that are found in raw materials. This condition has been accentuated the past months because of the necessity of using oils and fatty acids from widely scattered sources. Recently, even chemicals which have been uniform for many years have shown variations which, if not compensated for in the formula, will cause untold difficulties and many re-worked batches. An accurate evaluation of raw materials in advance of their use is absolutely imperative, and formulae should be adjusted, thereby eliminating adjustments by guess during manufacturing, always an extremely costly procedure, but too often permitted.

Laboratory Control

Usually time periods of from 30 minutes to 10 hours are required to secure approval from the laboratory of a batch of grease. While sometimes preliminary testing is done on the greasemaking floor by the greasemaker himself, it is usually required that laboratory approval be secured before filling is started from any

batch, or before the batch is transferred to storage. It is, therefore, quite clear that many equipment hours are lost while such laboratory approval is being secured and that many manhours are also lost while filling crews are standing by awaiting release to start filling from any particular kettle. It is our recommendation that in each case a study of this situation be made, looking toward speeding up the laboratory control cycle. If the greases are manufactured with chemical precision as outlined above, then in our opinion there will be such a uniformity of product that many of the laboratory tests which are now made can be eliminated. In fact, it is possible to package greases immediately after their manufacture is completed without full laboratory check, provided that chemical control and uniformity of manufacturing procedure disclose, as they have in other cases, that the number of off-specification batches is so small as to be insignificant. If the number of off-specification batches per year can be kept very small, then the loss to be encountered should an off-specification batch be packaged, is insignificant by comparison to the cost in equipment hours and manpower of awaiting full laboratory approval.

(Continued on following page)

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Use of Labor

It has been our purpose in studying greasemaking operations to reduce the number of hours per week in which actual greasemaking takes place to five 8-hour days, if possible, whereby the number of employees which it is necessary to have who are skilled in greasemaking will be greatly reduced. To produce the volume of greases required in five 8-hour days per week is entirely feasible, provided some additions are made in the equipment available and provided strict rules and regulations for chemical and manufacturing control are imposed under proper continuous supervision. Thus, the reduction in manpower required will result in large savings per annum in greasemaking operations.

Filling and Packaging

In most grease plants, filling and packaging operations require from 2-16 hours, depending upon the size of the packages. As stated above, packaging crews lose much time by standing by until laboratory approval is received. We believe the time cycle of 2-16 hours for packaging to be entirely too long and when this is the case an immediate study should be made, looking toward minimizing this time. If new or additional equipment is required for simultaneous operation of parallel packaging units, this equipment should be designed and built, or purchased. If it is necessary to modify the conveyor systems for the handling of empty containers and filled packages, this phase should constitute a study as well. A reduction in the time of filling and a regular schedule of filling will materially reduce the cost of these operations.

Conversion to Short Time Cycle Operation

It is well enough to point out why greasemaking is a science not an art, but many of the desirable features which have been mentioned are contingent upon availability of adequate equipment and

technique for its use. In order to make possible manufacture of precision batches in short time cycles, three elements of plant are imperative. They are:

1. Equipment for weighing *all* ingredients.
2. Adequate pressure saponification mixing equipment.
3. Fast high temperature heating means.

Attached to this paper is a flow sheet which shows how these features may be applied to an existing grease plant. They are all in use in the Jesco Lubricants Company plant in North Kansas City, Missouri, and have been for over 16 years. The technique used in Jesco's plant is one which has been developed practically and one which is in daily use, as later typically described.

On the assumption that most grease plants are now equipped with double stirrer paddle type grease kettles, the following steps are necessary to convert any plant to this type of operation:

1. Select from existing paddle type equipment the necessary kettles for incorporation in the new system.

Since the time cycles are short, for example, less than 2½ hours for soda fibre greases, and 3½ hours for cup greases, usually only a small part of the available paddle type kettles is required. However, these must be adequately jacketed for high pressure high temperature heating.

2. Install pressure saponification and finishing equipment, oil heated, a design which provides extreme intimacy of mixing, high inter velocities, and consequently, very rapid heating.
3. Install a flexible circulatory system of heating for providing the necessary heating medium at high enough temperature levels to make short time cycles possible.

In the Jesco plant, these requirements are fully met. The plant is equipped with

One Stratco design atmospheric pressure, double stirrer paddle type kettles jacketed for oil heating, having capacity of approximately 15,000 lbs.

Two Stratco Pressure Contactors approximately 4,000 lbs and 2,200 lbs capacity, respectively, each equipped with

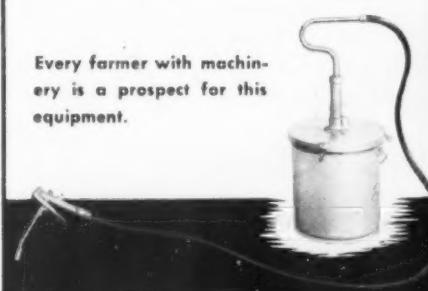
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The equipment as specified manufactures all of the products sold by Jesco and is capable of manufacturing approximately 15,000,000 lbs. of products a year in five 8-hour days per week. Approximately 200 different products are made to complete the line, including E. lubricants, soda and calcium base bases, aluminum greases, cutting oils, etc. The pressure contactors are of the high dispersion type with mixing effect provided by a high capacity impeller which turns over the total contents of the vessel many times each minute. Any and rapid changes in direction and velocity, as well as inherent shearing effects, provide the mixing and simultaneously provide the high velocities required for high heat transfer rates. The intimacy of contact provided in these contactors makes possible extremely short time cycles and high yields based on fatty acid, in fact yields from 10% to 20% higher than those being obtained by conventional soap making equipment.

A typical contactor is shown on the attached flow sheet.

The oil heating system is unique. It is small, compact and very efficient. It consists of a small inside fired radiant section, a tubular convection section and a plate type air heater with forced draft combustion. The overall thermal efficiency is over 90% and the radiant heat transfer rates average approximately 100,000 BTU/sq. ft./hour with rates in the hottest portion in the neighborhood of 250,000 BTU/sq. ft./hour. The heater is no masonry and can, therefore, be put down instantly when desired, without any concern about absorption of residual heat from masonry, usually an annoying problem. Likewise, the heater can be quickly fired to operating temperature.

A high flash oil is used as a circulating medium heated to 470°F—500°F. No trouble whatsoever has been encountered with this system in 16 years of operation, in fact, the heater has never been cleaned in that period of time.

The extremely high transfer rates are made possible by the fact that the circulating oil is jetted at very high velocity (approximately 100 F/S) against the sheet, which provides low film temperatures, high cleansing effect and very low deterioration of the heating oil. In fact, the heating oil used is reclaimed aircraft engine oil, which is used for ap-

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proximately one year and is then utilized in cheaper grades of grease. In a refinery, of course, the necessary heating medium can easily be removed from a process unit, used for a period and then returned to the unit with practically no cost. Oil is consequently preferred as a heating medium by most refinery operators.

The heating system has another very important feature. It provides a closed circulating system, including a reservoir of hot oil which makes possible the withdrawal of any desired quantity of heating medium to as many different points as desired, without in any way influencing the velocity of the oil through the heater. To make the system absolutely fool-proof, separate pumps can be provided for circulating oil through the heater and to the jackets of the vessels to be heated. The same circulating system can be used for suction heaters on storage tanks, for tank car heating and for any other required heating in the grease plant. Steam for tank heating is, therefore, not required. In cases where a large number of kettles and other facilities must be simultaneously heated, parallel heaters should be considered as a protection against shocking circulating oil temperature.

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Steam Versus Oil Heating

Most grease plants are equipped for use of steam as a heating medium. If high enough pressure steam is used to provide an adequate temperature head for rapid heating with resultant short time cycles, then the jackets of the vessels must be designed and built for extremely high working pressures. This is not desirable and is expensive. When circulating oil is used as a heating medium, the jackets are all low pressure jackets and mechanical construction is relatively easy and cost is lower.

Typical Process Cycle

In this paper, we obviously cannot begin to describe the multitudinous techniques that are employed in manufacturing a wide diversity of products. We do feel, however, that it will be of general interest to describe a typical manufacturing cycle. Since soda fibre greases are a product common to most greasemakers, we shall give as an example the procedure which is followed in the manufacture of, let us say, a No. 1 Soda Fibre grease. For this purpose, the two pieces of apparatus used are the 4,000 lb. pressure contactor

and the 15,000 lb. paddle type kettle. The contactor is charged with a weighed amount of fatty acid or fat and is filled to its 4,000 lb. level with the hydrogenated carbon oil to be used. The contactor is started and heat is applied until the temperature reaches approximately 190°. At this temperature, the dry powdered or flake caustic soda and a weighed quantity of water are added to the contactor. The vessel is sealed and the temperature is lifted as rapidly as possible to a temperature of approximately 370° F. It has been found that in every case of manufacture in this type of equipment the time of heating is the bottleneck and by the time the intimate mixture reaches a temperature of 370° F and a pressure of approximately 70 p.s.i., the soap is always completely manufactured. The time required for charging a manufacturing of soap never exceeds 15 minutes for any kind of soap. Since heating is the bottleneck, recent designs of contactor equipment have materially increased the heating surface available as to further reduce the time of manufacture. It is very interesting to



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to be carefully noted that the soaps manufactured in this manner are fluid and are not the gummy, sticky masses which are usually found in conventional operation.

While the soap is being made, a low grade of hydrocarbon oil is weighed into the paddle type kettle as a temperature of approximately 220°F. The hydrocarbon oil which is in the contactor, plus the amount which is weighed into the paddle type kettle, usually represents about 1/3 of the total hydrocarbon oil required to complete the batch. As soon as the contactor reaches a temperature of 370°F and a pressure of approximately 10 p.s.i., a large line is opened through which the soap-hydrocarbon oil mixture is transferred to the oil in the paddle type kettle. As the soap is transferred, much of the water contained therein is flashed off, and experience has shown what the moisture content of the soap-oil mixture will be, after flashing, to within hundredths of 1%. If a pressure paddle type kettle is available, the moisture content which the soap flashes can easily be controlled by varying the flashing pressure.

After the soap is transferred into the kettle which is in the paddle type kettle,

heat is applied to the paddle kettle for purposes of complete dehydration. Since the total quantity of material in the paddle kettle represents approximately 40% of the total batch, it can be readily seen that a relatively short time is required for this dehydration step, particularly since it is accomplished with oil heating at a high temperature level. It is usually necessary to heat the contents of the kettle to approximately 370°F in order to substantially complete dehydration. The dehydration step usually requires from 30 to 40 minutes. This operation can, of course, be speeded up by application of vacuum to the paddle kettle while heating, although this procedure cannot be accomplished in the Jesco plant because the paddle kettle was not designed for vacuum operation. After the dehydration step is complete, the remainder of the hydrocarbon oil is weighed into the paddle kettle at such a temperature that the entire mass will be in the order of 190°F, which is the temperature used for packaging. The oil is added very rapidly, approximately 10 to 15 minutes being required for this operation. Meanwhile, the paddle kettle is operating, giving a cursory blending to

(Continued on page 20)

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METALLIC SOAPS for GREASES

• By S. B. ELLIOTT Delivered before the 15th Annual N.L.G.I.
Convention, Edgewater Beach Hotel, Chicago, Illinois, Oct. 16-18, 1947.

Mr. Stanley B. Elliott

Mr. Stanley B. Elliott has had considerable experience in the chemical business and especially along the line of metallic soaps used in additives for lubricants.

He was graduated from Western Reserve University in 1939.

He conducted research on a wide range of metallic soaps for the Harsbaw Chemical Company.

In 1941 he joined Ferro Chemical Corporation where he has directed research activities on various metallic soaps used as additives, paints, varnishes, printing ink driers and fungicides.

He is the author of "The Alkaline Earth and Heavy Metal Soaps," a monograph published by the American Chemical Society.

During the war he directed research on aluminum soaps for the National Defense Research Committee and for the Chemical Warfare bomb and flame thrower fuels, and portable field equipment for continuous production of aluminum soap gels for incendiary purposes.

He holds a number of patents and patent applications covering techniques, equipment, and chemical agents used in metallic soap manufacture.

He is a member of the American Chemical Society, American Society for Testing Materials, American Wood Preserver's Association, and the Society of Cosmetic Chemists.

At present he is active in directing the development and manufacture of items of interest to the petroleum industry, such as metallic soaps, organometallic compounds, and various metal reaction products.

In any discussion of metallic soaps suitable for the manufacture of greases, it is generally necessary to consider specific metals and organic acids because of the great variations among different soaps. However, before individual soaps are discussed, it is believed valuable to consider the general characteristics of soaps and soap-hydrocarbon systems. For the purpose of this paper, metallic soaps are considered as compounds formed by replacing the cation, the acid hydrogen or its

equivalent, in a complex, monobasic organic acid by a metal. Though the means by which this is accomplished may vary, the end result is the same. Mono-, di-, tri-, or tetravalent metals may be involved in the reaction, in which case, provided the normal salts are formed, one, two, three, or four hydrogens respectively are replaced from as many acid molecules.

Though it is possible to prepare very pure metallic soaps for academic purposes, economic considerations demand that the soaps used in the manufacture of greases be based on fatty acids or fats of extremely variable composition. Thus, though the metal oxides or hydroxides used may generally be readily procured in a highly purified form, the fatty materials are usually subject to the wide variations common to natural raw materials. These circumstances, of course, make substantially more difficult the problem of securing reproducible gelation. Thus, it is believed of interest to review the effects of the various raw materials on the metallic soap and metallic soap-hydrocarbon system. The first materials to be considered are fatty materials, the source of the anion in the final soap.

Fatty Materials

Though rosin derivatives are important, soap-forming materials are generally the fatty acids or their glycerides and are derived from vegetable, animal, or fish sources. The source of the fatty material is of little consequence, however,



S. B. Elliott

the important matter being the types of acids which are present. Thus, it is no consequence whether any particular acid has been introduced into the final soap from corn oil or from lard oil as long as the proper quantitative balance is maintained, though incidental impurities typical of some fat sources can exert powerful influences through peptization.

Both saturated and unsaturated acids are usually present in ordinary greases and these acids are virtually always more

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carboxylic. The chain length of the important acids present, an important factor in determining the gelation characteristics of the soap, vary from approximately 14 carbon atoms to as high as 22. The latter is rather high and such acids are usually not found in high concentrations.

The melting point of the saturated acids increase from 54° C. for myristic (C_{14}) to 82° C. for behenic (C_{22}). More important, however, is the increase in hydrocarbon solubility of the metallic soaps as the chain length increases.

Unsaturated Acids

The unsaturated acids, belonging to a series of varying degrees of unsaturation, are more complicated since here too the chain length varies, though not usually over as broad a range. The melting points of the unsaturated acids are generally lower and the solubility of their metallic soaps greater than those derived from saturated acids of comparable chain length.

The soaps of some unsaturated acids are generally required in a grease in order to secure adequate hydrocarbon solubility, however, because unsaturation is always

accompanied by instability toward oxidation, it is desirable that the number of double bonds per molecule be kept to a minimum and conjugated double bonds, especially sensitive to extensive oxidation, be kept to a minimum. Thus, such an acid as oleic is most suitable from the standpoint of soap solubility as well as stability.

The induction period of an unsaturated acid, of course, is not a function of the degree of unsaturation, but rather of the concentration of natural inhibitors present, but once the induction period has been exceeded the amount of deterioration is much greater in the case of highly unsaturated acids.

(Continued on page 13)

"TECHNICAL COMMITTEE COLUMN"

(Continued from page 3)

Dr. J. C. Geniesse, The Atlantic Refining Company.
Mr. N. J. Gothard, Sinclair Refining Company.
Mr. Gus Kaufman, The Texas Company.
Mr. R. R. Matthews, Battenfeld Grease & Oil Corporation.
Mr. T. G. Roehner, Socony-Vacuum Laboratories.
Mr. N. D. Williams, The Pure Oil Company.
Mr. W. A. Wright, Sun Oil Company.

The following is a list of their projects which are currently active:

1. Low Temperature Torque Tests.
2. High Temperature — High Speed Testers.
3. Determination of Dirt Count.
4. Screening Test Machines.

All of the above projects concern only greases for anti-friction bearings. The scope and status of each project will be covered in later issues of this column. However, it should be emphasized now that none of the above projects represent duplication of effort or conflict with activities of other organizations, such as ASTM Technical Committee G, working on grease projects.

Finally, we would like to repeat the request included in the announcement of this column in the December 1947 issue of The Institute Spokesman, namely, that contributions will be very welcome and may be addressed to Mr. T. G. Roehner (Socony-Vacuum Laboratories, 412 Greenpoint Avenue, Brooklyn 22, N. Y.), Mr. H. L. Moir (Pure Oil Company, 35 East Wacker Drive, Chicago 1, Illinois), Mr. William H. Oldacre (D. A. Stuart Oil Co., Ltd., 2727 South Troy Street, Chicago 23, Illinois), Chairman and Vice-Chairmen, respectively, of the Technical Committee.

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President's Column...

All bearings, to operate efficiently, must have a continuous film of proper lubricant. This, of course, is an undeniable fact. Consequently, lubricating grease manufacturers build their products to a specification to provide for proper lubrication under all temperatures.

But temperatures, as we all know, fluctuate. True, there are four seasons. But there is nothing constant about the temperatures in any given one. In most parts of North America, summers are usually warm and winters cold. But many times we have a cold snap in summer; or a spell of warm weather in winter. And these weather changes are apt to occur with surprising suddenness —



J. R. Corbett
President N.L.G.I.

especially in so many parts of the country where the weather is "variable."

When we think of lubrication, therefore, we've got to do more than determine the right kind of lubricant for the machinery in question. We've got to make sure that the lubricant is properly dispensed to the proper place. Because the finest lubricant in the world is useless, unless it's dispensed properly.

Extreme weather conditions — especially freezing temperatures — play havoc with the dispensing operation. Under these conditions correct lubrication is often sacrificed to meet dispensing requirements.

When adverse weather conditions do exist, and the user runs into dispensing difficulties, he is apt to blame the grease manufacturer for his problem. Actually, it is not the grease manufacturer's fault. It is difficult to work out a lubricating product that will dispense easily under the varying degrees of temperature change. Nor is the blame for dispensing difficulties to be shifted to the dispensing equipment manufacturers. They have done a magnificent job of providing the

industry with the very best of modern scientific equipment.

Where an extreme weather condition exists, it is often difficult for the user to keep his dispensing equipment in normal temperatures, away from dirt and foreign substances. *But this should be done if at all possible.* In this way, with dispensing equipment functioning properly, correct lubrication is a matter of course. And correct lubrication means less maintenance cost on the part of the operator.

Users are urged, where dispensing equipment must, of necessity, be kept and operated in abnormal temperatures to make this fact known to their supplier. In this way, they can have a special lubricant made to dispense properly under the existing conditions, which may sometime call for a sacrifice in correct lubrication.

And, finally, here is a word of well-meant advice to the user: Proper care of dispensing equipment will avoid many lubrication difficulties that might incorrectly be attributed to the deficiency of the lubricant or the dispensing equipment.

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METALLIC SOAPS FOR GREASES

(Continued from page 11)

In Sett greases the rosin acids and associated terpene compounds are not stable to aerial oxidation so that these materials are especially susceptible to rapid gumming.

Metal Compounds Used for Soap Manufacture

Though everyone in the grease industry is familiar with suitable metal compounds to be used for the manufacture of various metallic soaps for greases, it is interesting to consider the factors which may lead to unsatisfactory performance. Of the factors operating, one group may be said to be related to the nature of the metal which forms the soap and the other concerns metals present as impurities.

Of the soaps of calcium, sodium, uranium, aluminum, lithium and lead, only lead can be said to be a metal which, because of its catalytic activity, imposes special problems in lubricant compounding. Though the other metals have been reported at various times to be oxidation or polymerization catalysts, their activity is low. Lead, however, is a rather effective polymerization catalyst, so that there is a strong possibility that

the unsaturated acids may be rapidly degraded.

Polymerization and oxidation of the unsaturated anions of metallic soaps almost invariably leads to a modification of the physical properties of the soap, of which hydrocarbon solubility is one of the more important, so that a substantial drift in the properties of the lubricant, of which the soap is a component, occurs. Because of the undesirable nature of this phenomenon, there has been a pronounced tendency to eliminate unsaturated acids from compositions involving lead.

The effect of metallic impurities was mentioned above as having an important bearing on the performance of many greases. This situation obtains because many greases operate at elevated temperatures in the presence of dissolved oxygen, so that any metals which may function to promote oxidation may lead to accelerated deterioration. Once again this deterioration is, in large measure, a matter of the breakdown of the desired grease structure because of the change in physical properties of the metallic soap's physical properties.

The unsaturated acids, comprising the anions of a portion of the soaps present in a typical grease, possess finite induction



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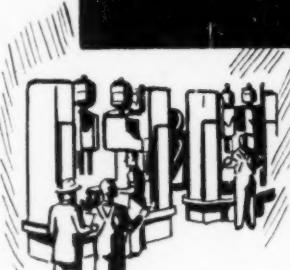
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periods by virtue of the natural antioxidants present. The presence of small quantities of catalysts, and iron soaps are highly active, can drastically shorten this period after which there is a rapid rise in the peroxide value of the unsaturated acids present and a drop in the iodine value. Subsequently, of course, the peroxide value decreases because of peroxide decomposition, but the physical properties of the soap have by then changed markedly and usually so has its thickening power, both because of molecular change and the development of peptizing agents incidental to long chain decomposition.

Thus, the presence of metallic soaps of metals having high catalytic activity must be restricted closely, for even antioxidants operate under difficult circumstances in the presence of an active positive catalyst such as an iron soap.

Lubricating Oil for Greases

Since a grease is used primarily for the lubricating oil which it contains, it is obvious that the oil must be of satisfactory characteristics so far as oiliness, viscosity index, viscosity, volatility, etc., are concerned. However, from the viewpoint of a grease as a hydrocarbon-me-

tallic soap system its solvency characteristics are of extreme importance.

Aliphatic hydrocarbons have been shown to be the poorest solvents, naphthenic hydrocarbons possessing substantially higher solvency. Thus, since we are primarily interested in the metallic soaps used in the system, it can be said that the anion of the soaps used to thicken paraffinic hydrocarbons must possess a structure conducive to higher solubility than those which are tolerable in a naphthenic hydrocarbon system.

Since the hydrocarbon appears to interact with the soaps in some instances, possibly partially dissolving the nonpolar portion of the soaps, higher-solvency oils in many instances are useful when the soaps possess limited solubility.

Under many operating conditions the viscosity of the base oil appears to be an important factor since the high shear rates ordinarily encountered break many of the bonds between soap aggregates, the viscosity of the mass approaching that of the oil.¹ However, on reversion to low shear or static conditions, the soap content appears to control the magnitude of the yield value. At intermediate points

1. Blott, J. F. T., and Samuel, D. L., Ind. Eng. Chem., 32 (1), 68 (1940).

between very high shear rates where the soap-particle linkages are few in number and low rates of shear, there exists an interesting field of investigation to determine the effect of nonpeptizing solvents other than the usual ones used, for some materials are known which change quite markedly the slope of the "viscosity rate of shear" curves for soap-hydrocarbon systems.

Manufacturing Methods

No doubt everyone in the grease industry is familiar with both fusion and precipitation processes for preparing metallic soaps. The fusion process involves reacting a metal oxide, hydroxide, carbonate, or similar compound with either an acid or the ester of an acid in an essentially nonaqueous medium so as to form the soap and water, glycerol, or similar material.

The precipitation process consists of first preparing a water-soluble salt of the desired acid and then precipitating the water-insoluble metal soap by adding the proper water-soluble metal salt. The metallic soap is then washed and dried.

Both the fusion and the precipitation processes and the products they produce can be varied widely by modification.

(Continued on page 16)



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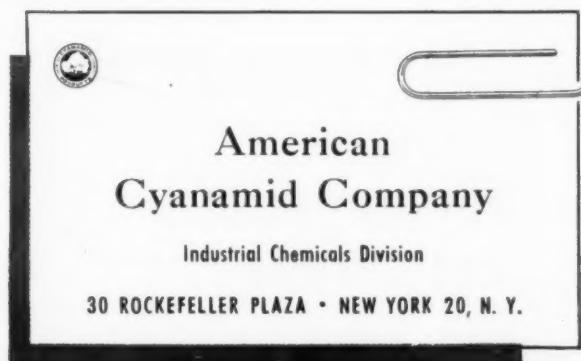
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METALLIC SOAPS FOR GREASES

(Continued from page 14)

time, temperature, type of reactants, reactant ratios, etc., and it is in these things we are interested.

Metallic Soap-Hydrocarbon Systems

Before discussing any particular metallic soaps, a general review of the ways by which metallic soaps may thicken hydrocarbons is considered useful. Though there are still great gaps in the information available, there is enough known to be useful in our consideration of specific soaps.

Hydrocarbons may be thickened by metallic soaps in different ways. First, we may have gels, which are semi-opaque systems containing noncoherent lumps. If the concentration of soap is high enough, the soap crystals will entirely occupy the system. The degree of crystallinity, of course, varies greatly depending on the soap, aluminum soaps representing extremely fine crystals while sodium soaps may form very large crystals. Furthermore, whereas some greases will consist of gels of crystalline particles in oil under most conditions, others will be true jellies. Jellies are rigid and elastic systems which are optically clear in the

absence of impurities and finally sols are the free flowing liquid form and they also are clear.

There is no sharp differentiation between jellies and sols, for the former appears to consist of a continuous medium in which colloidal particles are suspended and loosely bonded so as to develop a brush heap structure and the latter is much the same except that there are fewer bonds and so the resistance is much less.

These transitions may be seen when a suitable metallic soap is placed in an acceptable hydrocarbon. When placed in the liquid, the soap continues to swell until the particles reach a maximum size, the particles remaining discrete unless compression or long contact causes coalescence. If the material is sufficiently amorphous, or if the temperature is raised, then additional swelling occurs and the mass passes through the jelly to the sol stage.

Once the symmetrical molecular arrangement of a crystalline soap has been disturbed by heating, for example, so that the system has passed from a gel into a jelly or sol there will be a more or less slow reversion to the original gel structure, but this will surely occur if the gel phase is the stable phase at ambient tem-

peratures. This, of course, requires the grease manufacturer that he adjust the concentration of soap or the type of soaps used so that a spontaneous phase change does not occur on storage.

As noted, the reversion is sometimes a slow process, because of the random distribution of molecules, but it may occur much more rapidly if the system was not heated far above the formation temperature of the jelly. Thus, apparently invisible residual gel particles may act as foci for rapid reversion to the state.

The phase diagrams for various metallic soaps in the same solvent vary widely as would be expected, and different types of solvents sometimes affect the diagram markedly. Further, though some soaps are ordinarily used in the anhydrous state, others such as calcium soaps are known to form hydrates and it is sometimes the hydrated form which forms systems of acceptable physical qualities. Thus, the development of acceptable lubricating oil-soap systems is very much an empirical matter.

Peptizers

The term peptizers, as commonly used in the industry, can be said to refer to any compounds which will cause the

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ral of thickening or gelatinization or regulation of grease systems.

A great amount of work has been carried out on metallic soaps by various companies, with the object of establishing some basis for activity so that these association colloids could be modified as to the degree of association. Some have been interested in higher viscosities, some lower, but control was desired in every case.

The research activities of our own staff were early directed toward the possibility of predicting activity from dipole moment measures since the correlation of high-dipole moments with high peptizing activity is very good. And here, of course, the dipole moment referred to is the number indicating the magnitude of asymmetry of the molecule. However, examination of a large number of compounds has indicated the correlation accidental, the most conclusive evidence resulting from evaluation of the activity of compounds of the same chemical structure but having different dipole moments.

Apparently peptization consists of the opening of the bonds between adjacent articles, the severed linkages then being satisfied by solvent or the peptizer. Thus, chemical functions vary much as tem-

perature does, a transition often being noted from gel to jelly and from jelly to sol, depending on the amount added. Since, for a given soap concentration in a number of hydrocarbon-soap systems there is often an increase in viscosity when passing from the gel to the jelly phase, the presence of a certain amount of peptizer can be important to the grease compounder. Since, however, the passage from jelly to sol state is accompanied by a marked viscosity reduction, it is important that the peptizer concentration be controlled closely.

There appears to be a distribution of peptizer between the soap and hydrocarbon so it is usually desirable that the peptizer be somewhat soluble in oil. At the same time, it must have some affinity for the material to be peptized since it probably interacts with some of the groups exposed on the soap aggregates, thus freeing them from each other. Such interaction or sorption is, of course, favored by a limited solubility.

Syneresis

Syneresis, or the sweating and bleeding of oil from a grease is, of course, intimately associated with the metallic soaps used to thicken the system. Primarily, it reflects the slow adjustment of a meta-

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stable hydrocarbon-metallic soap system is making in order to establish stability. As would be expected, low concentrations of soap, low oil viscosity, and high pressures applied in certain ways are factors which will cause oil to separate from the system.

Though the physical chemistry of greases is certainly more complex than these brief comments on metallic soap-hydrocarbon systems would indicate, the most important phenomena have been mentioned as a foundation for our consideration of individual soaps, the first being those of aluminum.

Aluminum Soaps

Though greases based on aluminum soaps are not nearly as important volumewise as are those based on other metals, the complicated nature of their structure and the complexity of the thickening action make aluminum soaps ideal for our first consideration. The problems arising incidental to their utilization are those encountered, in many cases, with other soaps, so analogies are useful.

Dr. J. W. McBain and his coworkers at Stanford appear to have done some of the best work in the field of aluminum soaps, having elucidated most of the known basic concepts of their activity, structure, and formation.

Most of the aluminum soaps manufactured are based on lauric, palmitic, stearic, or oleic acid or mixtures thereof. Though there are a large number of various products supplied, and though they are even designated as the mono-, di-, or tri-soaps in many instances, they appear to be empirical mixtures of the mono- and di-soaps together with free acid and acid complexes in every instance. Thus, a review of the behavior of these definite soaps will serve to explain the performance of the soaps of mixtures which are regularly available.

Composition

By reacting sodium soaps containing varying amounts of excess base with a solution of aluminum salt, or by comparable methods, it should be possible to precipitate the tri-soap, Al^3FA_3 , the di-soap, $\text{Al}^2\text{FA}_2\text{OH}$, and the mono-soap $\text{Al}^1\text{FA}^1(\text{OH})_2$. Actually, as already indicated, the tri-soap never forms but rather the di-soap precipitates, along with fatty acid ("FA").

The fatty acid present, however, is not usually free but is sorbed or bound to the soap. Depending on whether the composition of the precipitated soap is close to that of the mono-soap or tri-soap, the acid not combined with alumin-

um varies from very little to substantial quantities.

Gel Structure

The fatty acids not combined directly with aluminum act, of course, as peptizers. Thus, the total amount of such acid must be held to a reasonable level. At the same time, soaps too high in aluminum content cannot effectively be used for, though the uncombined acid is low, the content of mono-soap rises and the latter appears to be very ineffective as a hydrocarbon thickener.

Evaluation of the pure mono- and di-soaps confirms that the di-soap offers the best possibility of functioning as a successful thickening agent. However, the history of the soap, as it modifies the crystallinity of the material, appears to exert a strong influence upon its behavior as a thickening agent.

As mentioned before, there is a transition from gel to jelly to sol. Thus, for a given composition of soap the phase diagram will vary somewhat depending on the crystallinity. If the soap is highly crystalline, then gels, semi-opaque lumps, will form unless the temperature is taken adequately high. On the other hand, if the soap is amorphous, the dispersion temperature is substantially lower, a clear, rigid, and elastic jelly readily forming.

As the temperature rises, in either case, and thermal disruption of bonds occur, there is a transition to the clear, freely flowing sol.

Investigation has indicated that aluminum dilaurate is an association colloid in benzene,² and other work has indicated that aluminum soaps in general associate in hydrocarbons. The association increases rapidly with concentration, the osmotic pressure divided by the concentration decreasing rapidly with concentration. The association particle weights range from about 300,000 to 2,600.

It appears that this structural viscosity is caused by loose linkages and aggregations of colloidal particles. The linkages probably lead to a brush-heap arrangement which enmeshes and immobilizes substantial quantities of solvent. The association particles, furthermore, probably contain both solvent and soap and are not pure soap particles. A high degree of working serves to break many of the linkages so that a decrease in viscosity occurs. Under ordinary circumstances, however, the decrease is not too great, an equilibrium establishing itself between new linkages being formed and old ones being broken.

Since the solvent appears to interact with the soap molecules, the hydrocarbon

part of the soap conceivably being being in partial solution by the solvent, naphthenic solvents produce greater diffusion penetration than do paraffinic, other factors being the same. The penetration is also substantially affected by the cooling rate since the sol-jelly-gel transition requires a finite time because of the random orientation of the highly complicated molecules.

Aluminum-Sap Peptizers

Though all metallic soaps appear to be sensitive to one type or another of peptizer, aluminum soaps appear to be sensitive to peptization by water to an usual degree.

Most aluminum soaps have their genesis in aqueous systems, the water subsequently being removed. If the soaps are of exceptional purity, then we have only to consider the hydroscopic nature of the soaps themselves. In actual practice, sufficient inorganic salts are often left in the soaps to markedly accelerate sorption of water at relatively high humidities. Further, the soap itself rapidly sorbs small amounts of water where it appears to be held by surface forces. It is interesting to note that aluminum soaps appear to form no hydrates, a smooth sorption isotherm being obtained.³ Further, not much more than 3 per cent water appears to be taken up, even at very high humidities.

Besides the sorbed water, there may also be water formed by reaction of fatty acids and hydroxyl groups, but this action does not appear to occur to a substantial degree except at elevated temperatures.

Besides water, aluminum soaps are peptized by a number of other materials among them amines, phenols, and a number of polyhydroxy straight-chain compounds. As far as is known, these materials function much the way described previously.

3. Shreve, G. W., Pomeroy, H. H., and M. K. J., *J. Phys. Colloid. Chem.*, 51 (4), 963 (1947).

(Continued in next issue)



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2. McBain, J. W., and Working, E. B., *J. Phys. Colloid. Chem.*, 51 (4), 974 (1947).

New Members

Again it is our pleasure to welcome one of the new members who have joined the N.L.G.I. recently. All are Associate Members.

Gross & Co., Inc.

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Eugene W. Adams Asst. Vice Pres.

For the story of A. Gross & Company it must go back to Cincinnati in the year 1837. Cincinnati was then the country's center for the manufacture of lard, soap and candles. It was there, over a century ago, that Andre Gross founded the company which still bears his name.

Through the years, A. Gross & Company has supplied American Industry with one of its most versatile raw materials—fatty acids. In addition to the lubricating grease industry, fatty acids of both vegetable and animal origin are used in the manufacture of shaving soaps, cosmetics, synthetic paints, plastics, textiles and a myriad of other products. In order to locate in the center of the Eastern manufacturing area, A. Gross & Company moved from Cincinnati to Jersey City, N. J. and later to its present plant on Newark Bay, in Newark, N. J., where raw fats can be received by water as well as rail.

In this plant is a modern stainless steel distillation unit. Only recently, new equipment was installed insuring continued quality production which will keep the company among the principal leaders in the industry. It is a far cry from that of the original plant but the present Chairman of the Board of Directors, Mr. David Mahany, a grandson of the founder, still carries out his policies. Under his leadership the other officers are: Mr. Walter Hagedorn, President; Mr. Maurice J. McCarthy, Vice President and Treasurer; Mr. John B. Blum, Secretary.

A wholly-owned subsidiary, A. Gross Candle Co., manufacturers of all types of paraffin and beeswax candles, has recently started operation in a new and modern plant in Linden, N. J., a move from its older plant in Baltimore, Md.

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(Continued on following page)

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Vern I. McCarthy, son of P. H. McCarthy, who has been associated with his father in the business since his youth, is president of the organization.

George E. McMahon is vice president in charge of manufacture.

Dale M. Harpold is vice president in charge of general sales.

The company manufactures a complete line of steel shipping containers ranging in capacity from 1 through 15-gallon, in various Interstate Commerce Commission specifications. Sales offices are maintained in principal cities and when the availability of steel is normal, warehouse stocks are likewise carried.

It would be generally repetitious to refer to the acute shortage of sheet steel with its resultant elongation and protraction of a steel container manufacturer's scheduling, not to mention the inconvenience that is caused the users of steel shipping containers. The Vulcan organization is making a sincere effort to allocate and distribute steel containers in proportion to the steel available to their customers."

GREASE MANUFACTURE . . . AN ART OR SCIENCE

(Continued from page 9)

the oil and soap. As soon as the formula is complete, a circulation is started from the paddle kettle to the contactor, and back to the paddle kettle, during which time the contactor is in operation. Approximately 3 to 5 minutes contact in the contactor are required to thoroughly blend the soap and hydrocarbon oil. In fact, it is in the contactor that the grease is really manufactured. It is very interesting to note that the material being circulated is a fluid and, although the grease may have a melting point considerably higher than the circulating temperature, it has never taken its initial set and is consequently in the fluid condition and easy to handle.

At the proper moment in the circulation cycle, the product is withdrawn from the contactor directly to package. During the circulation, samples are withdrawn for rapid determination of hot penetration which is used as a manufacturing basis and a time saver. This

check is accurate because of correlations which have been worked out by experience between hot and cold penetration.

If it is desired, the finishing operation can be carried on batch-wise with contactor filled only to the level of top of the circulation tube with vacuum applied to the vessel. Such an operation simultaneously deaerates the grease before it goes to the package. The flow in the contactor is such that large areas exposed to deaeration and it, therefore, accomplishes this result very satisfactorily.

The entire time cycle consumed in operation like this is from 2 1/4 to 2 1/2 hours from charging of the soap kettle through packaging in large packages, it is possible, therefore, to manufacture three batches in one 8-hour day. This is a very important consideration when compared to the time cycles of 24 to 36 hours usually consumed in the manufacture of a soda fibre grease.

Batch Versus Continuous

During the past several years, there has been considerable interest in attempting to develop satisfactory continuous

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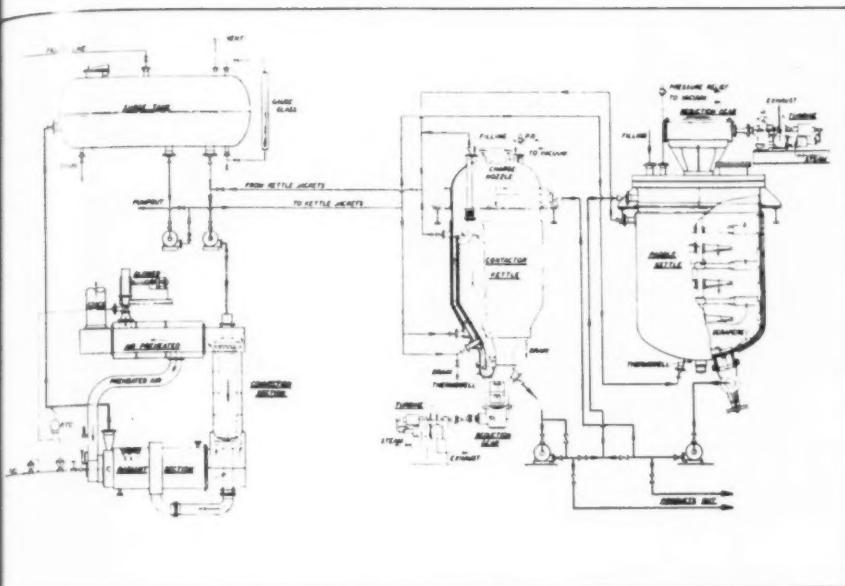
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greasemaking systems. It has usually been found that unless extremely large quantities of a single product are to be manufactured, continuous operation leaves a great deal to be desired because of contamination, difficulty of dehydration,

difficulty of controlling ingredients by volume, and several other factors. In fact, most greasemaking operations which are termed continuous are in reality batch plants with the oil-soap mixing step.

(Continued on following page)

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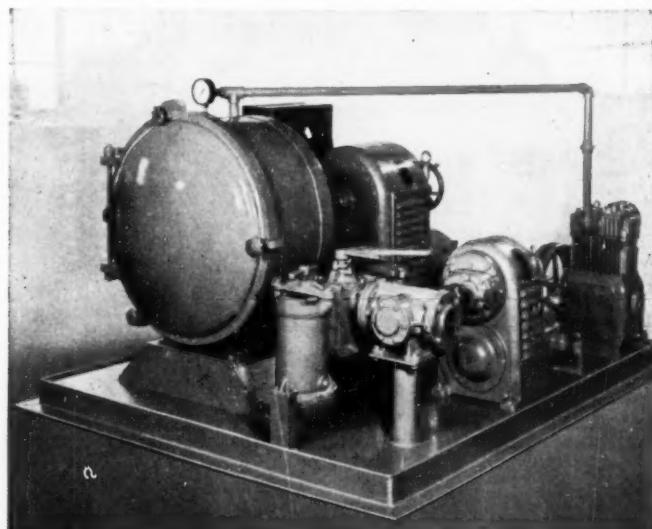
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Grease Homogenizer, showing feed pumps, strainers and vacuum pump.

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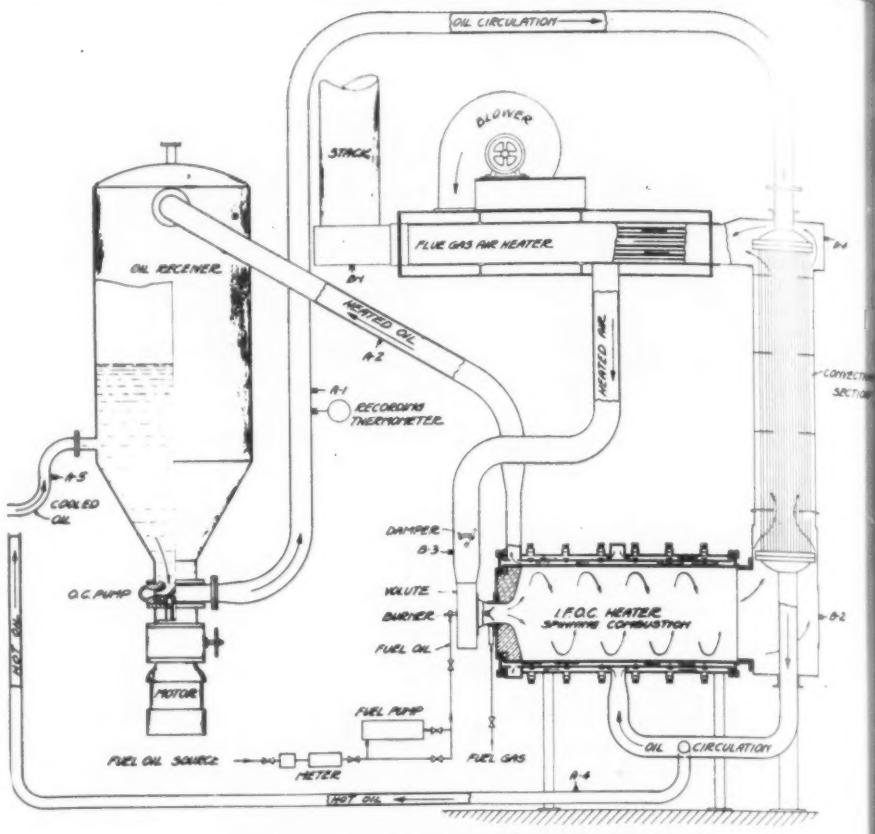
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only, being continuous. It is the writers' belief that much more flexibility can be had in any grease plant by short time cycle batch compounding of ingredients than by attempting continuous operation for the relatively small volumes of each product required.

In closing, we should like to state that grease manufacture can be simplified and modernized, and that manufacturing costs can be materially reduced by the application of relatively simple equipment to existing plants, and that, in our opinion, greasemakers, as a group, should convert their operations to a scientific manufacture of definite products of definite specifications by simplified and short time cycle techniques.

The authors of this paper will be pleased to answer any questions concerning the methods employed in the manufacture of the principal products, and to explain to you any of the construction details of the equipment, in response to any questions which you may care to ask.

We thank you.



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